UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service

PROBLEMS AND OPPORTUNITIES IN THE INDUSTRIAL UTILIZATION OF WHEAT

Northern and Western Utilization Research and Development Divisions
and Crops Research Division
with the
Technical Advisory Committee, Millers' National Federation, and
Technical Committee, Association of Operative Millers

at Peoria, Illinois
October 4, 1960

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PROGRAM

ABSTRACTS OF REPORTS

DISCUSSIONS

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FOREWORD

The annual Wheat Utilization Research Conference was held October 4, 1960, at the Northern Regional Laboratory under the sponsorship of the Northern and Western Utilization Research and Development Divisions and the Crops Research Division of the Agricultural Research Service, U. S. Department of Agriculture, with the Technical Advisory Committee, Millers' National Federation, and the Technical Committee, Association of Operative Millers. At this conference emphasis was placed on industrial utilization with presentations on performance requirements for expanding uses of products derived from wheat in the paper, textile, and adhesives industries, in addition to the progress reports on research on wheat in the cooperating Divisions.

The following Federal and State groups interested in research were represented:

United States Department of Agriculture
Agricultural Research Service
Washington, D. C. and,

Northern Utilization Research and Development Division
1815 North University Street
Peoria, Illinois

Western Utilization Research and Development Division
800 Buchanan Street
Albany 10, California

Crops Research Division
Beltsville, Maryland

Agricultural Marketing Service
Washington, D. C.
Market Quality Research Division
Market Potentials Research Branch

Kansas State University
Department of Flour and Feed Milling Industries
Manhattan, Kansas

Kansas Wheat Commission
Hutchinson, Kansas

Nebraska State Department of Agriculture
Lincoln, Nebraska

University of Nebraska
Department of Biochemistry and Nutrition
Lincoln, Nebraska

SUMMARY

At this wheat utilization conference reports were presented on the performance requirements of cereal-derived products for use in the paper, textile, and adhesives industries. Areas of fundamental and applied research needed on wheat products to supply these requirements were described.

The volume of wheat flour now being used industrially could be greatly expanded if simple means could be found to modify the starch and improve dispersibility of the gluten, so that the product could be used in traditional starch applications. Even more desirable would be the development of outlets not competitive with present starches.

An economical, acid-conversion process for modifying dry flour to meet viscosity requirements of tub-sizing and coating applications for papermaking has been developed. Products have performed acceptably in paper applications. Other acid-modified flours have been prepared for trial in textile and gypsum board uses.

Extensive evaluation of variously modified wheat flours for tub-sizings and coating adhesives in papermaking has shown them to be equivalent to some of the best commercial starch-based agents.

The preparation of low-protein fractions by fine-grinding and air-classifying six commercial unbleached, straight-grade flours showed low protein content of fractions inversely related to hardness of the wheat. All soft wheat flours yielded fractions containing not over 3 percent protein; the lowest, 1.4 percent protein, was from Pacific Northwest soft white wheat. The minimum from hard wheat flour was a 4.9 percent protein fraction from a hard red winter.

The technology of starch usage in papermaking has not kept pace with either the intensive development programs in paper and paperboard or with research by the chemical companies producing products competing with starch. Major uses of starches in the paper industry for which specific properties are desired include the following: beater adhesives, pigment coating adhesives, surface sizing of paper and board, laminating adhesives, corrugating adhesives, and gluing adhesives. Properties for these various uses include a lower viscosity at high solids concentration, better water release, greater adhesion at low viscosities, more flexibility and elasticity of films, viscosity properties similar to latices, better ink receptivity, and starch-pigment film insolubility.

Four major uses of starch in the textile industry in order of decreasing importance are: warp sizing, home and commercial laundries, stiffening and firming agents in finishing fabrics,

and in printing pastes. Starch is losing out particularly to synthetics in printing pastes and easy-care finishes because of differences in performance, and there is stronger competition from synthetics for finishing agents in commercial and home laundries as a result of aggressive merchandising and convenience appeal. Losses of markets for starch as warp sizing are almost entirely due to pollution problems in disposal of plant effluents containing starch wastes. Solving the BOD problem will be necessary to protect these markets and might open up new ones.

The recent great expansion in use of adhesives is expected to continue. Cereal-derived adhesives have one major advantage--low cost, and one major disadvantage--low water resistance. Additional improvements are needed, such as in bonding strength, coverage, setting speed, tack, solids content, and flexibility, to greatly broaden the use areas outside of the paper industry. Possibilities for accomplishing this include: crosslinking with formaldehyde alone or in combination with urea, dimethylolurea, phenol, resorcinol, etc. or acrolein; incorporating water-resistant groups; and hybridizing the molecule with fatty acids or protein hydrolysates.

Recently developed techniques, particularly countercurrent extraction, silicic acid column chromatography, and gas chromatography, have made possible the separation of simple and complex lipids from wheat flour and the further segregation of five classes of complex lipids. Separated were a lipoprotein, galactolipids, phosphatidyl-ethanolamine and phosphatidyl-choline. In air-classified flour fractions total lipid content increased with total protein content.

A flour prepared from Pacific Northwest soft wheat was air-classified and analyzed for albumin and globulin proteins. High-protein fractions were lower in soluble protein than low-protein fractions, but the ratio of albumin to globulin proteins was about the same in the original flour and in the fractions.

Evaluation of wheat varieties has attained new precision--bread, cake, and cookie baking tests; improved micro tests; and basic knowledge of inherited differences combine to give earlier and better estimates of varietal potential.

Starch gel electrophoresis in the presence of concentrated urea has been applied for the first time to the separation of wheat gluten proteins. This new method revealed eight components in a gluten preparation, two of which had not previously been reported. Nine components were found in a water-soluble fraction (albumins and globulins) of a gluten preparation when examined by this method. Application of the method to comparison of proteins in different varieties of wheat is expected to reveal fundamental differences which may be related to gluten performance.

The unique viscoelastic properties of gluten reside in its α_1 component, the fastest-moving electrophoretic fraction. Sedimentation analysis in an aqueous system containing 0.03 M ammonium hydroxide and M urea showed that α_1 gluten was a mixture of sedimenting species with molecular weights ranging from about 50,000 up into the millions, averaging 1.6 million. Cleaving the disulfide bonds with performic acid at 0° C. reduced it to a single sedimenting species with a molecular weight of 21,000 and destroyed its viscoelastic properties.

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Agenda for Conference on
PROBLEMS AND OPPORTUNITIES IN THE INDUSTRIAL UTILIZATION OF WHEAT
Peoria, Illinois
October 4, 1960

8:00 a.m. Registration

Morning Session

8:15 a.m. Introduction
F. R. Senti, Director, Northern Division

8:45 a.m. Industrial Uses of Wheat Flour and Problems Involved in Their Expansion
H. H. Schopmeyer, International Milling Company

9:15 a.m. Preparation and Properties of Chemically Modified Cereal Flours
J. C. Rankin, Northern Division

9:35 a.m. Evaluation of Modified Wheat Flour in Papermaking
C. R. Russell, Northern Division

9:55 a.m. Preparation of Low-Protein Fractions from Soft and Hard Wheat Flours by Fine Grinding and Air Classification
V. F. Pfeifer, Northern Division

10:15 a.m. Recess

10:30 a.m. Performance Requirements for Expanding Uses of Cereal-Derived Products in the Paper Industry
J. W. Swanson, Institute of Paper Chemistry

11:00 a.m. Performance Requirements for Expanding Uses of Cereal-Derived Products in the Textile Industry
H. Y. Jennings, School of Textiles, North Carolina State College

11:30 a.m. Performance Requirements for Expanding Uses of Cereal-Derived Products in the Adhesives Industry
Irving Skeist, Skeist and Schwarz, Inc.

12:00 Noon Group Photograph (Assemble at Building Entrance)

12:15 p.m. Luncheon

Afternoon Session

2:00 p.m. Lipids and Lipoproteins in Wheat Flours
G. O. Kohler, Western Division

2:15 p.m. Distribution of Albumin and Globulin Proteins in Air Classified Fractions of a Soft Wheat Flour
J. W. Pence, Western Division

2:30 p.m. Fundamental Aspects of Wheat Variety Evaluation
L. P. Reitz and C. E. Bode, Crops Research Division

3:00 p.m. Separation of Gluten Proteins by Starch Gel Electrophoresis
J. H. Woychik, Northern Division

3:15 p.m. Effect of Disulfide Bond Splitting on Molecular Size of α_1-Gluten
H. C. Nielsen, Northern Division

3:30 p.m. General Discussion

3:45 p.m. Concluding Remarks
C. L. Mast, Jr., Millers' National Federation

ABSTRACTS OF REPORTS AND DISCUSSIONS

Introduction

F. R. Senti, Director
Northern Utilization Research and Development Division

The conference was opened with Dr. Senti welcoming members of the Technical Advisory Committee, Millers' National Federation; members of the Technical Committee, Association of Operative Millers; and guests.

In planning the program, discussions with Mr. C. L. Mast, Jr., Executive Vice President, and Dr. H. H. Schopmeyer, Chairman, Technical Advisory Committee, Millers' National Federation, led to the decision that this conference should emphasize opportunities and problems facing the increased industrial utilization of wheat. This decision did not reflect any desire to minimize the importance of research on food uses, indeed, it was planned to include progress reports on basic research which are important to both industrial and food uses.

Pulp and paper, adhesives, and textiles represent potential large consumers for industrial products based on wheat. In order to learn of the needs and opportunities for wheat products in these fields, an expert in each area was invited to talk on his specialty. A representative of the milling industry was selected to discuss the industrial utilization of wheat from the miller's viewpoint.

The basic and applied researches in the Regional Laboratories and Crops Research Division on utilization of wheat will be reviewed. Reports from the Northern Division will cover 3 main areas of research:

1. Basic work on composition and structure of gluten proteins.

2. Chemical modification of flour to enhance its industrial utility.

3. Separation of flour by fine grinding and air classification to produce fractions with improved food and industrial uses.

The reports from the Western Division will cover lipids and lipoproteins in wheat flours and the distribution of albumin and globulin proteins in an air classified flour. The Crops Research Division will report on fundamental aspects of wheat variety evaluation.

INDUSTRIAL USES OF WHEAT FLOUR AND PROBLEMS INVOLVED IN THEIR EXPANSION

H. H. Schopmeyer
International Milling Company

Substantial amounts of wheat flour are currently being used for industrial purposes; as glue extenders for plywood; core binders; adhesives; binders for charcoal and coal briquettes; in oil well drilling operations to remove the sludge; wallpaper paste; in the manufacture of starch and gluten, etc. For the most part, these uses require only low grade flour with very little chemical modification. Attempts to use wheat flour as a paper size, textile size, or as an adhesive, etc., have not been particularly successful due to difficulty in modifying the flour to get the flow and paste characteristics desired, also because of the tendency of the protein to separate in small chunks or curds during the sizing or gluing operations.

The volume of wheat flour being used industrially could be greatly expanded if techniques could be developed for readily modifying it to produce products comparable to dextrins, British gums, acid-modified starches, oxidized starches, etc., and the dispersibility of the gluten could be improved.

Much of the volume increase would be at the expense of pure starches which are now being used for these purposes. It would be most desirable to develop entirely new outlets for cereal products such as in plastics, insulating products, roofing materials, etc., which could grow to large volume businesses to benefit both Agriculture and Industry.

PREPARATION AND PROPERTIES OF CHEMICALLY MODIFIED CEREAL FLOURS

J. C. Rankin
Northern Utilization Research and Development Division

Earlier work at this Laboratory on derivatizing cereal flours with ethylene and propylene oxide improved flow and dispersion properties of flour pastes. However, they lacked certain other desirable paste properties such as reduced viscosities at high solids concentration required for many nonfood industrial uses. Work toward this objective has led to the development of an economical, dry, low-temperature, acid-conversion process for modifying flour to meet these industrial viscosity requirements. The nonaqueous method of conversion consists of treating dry flour with small amounts of acid at 25 to 35° C. for 1 to 8 hours. This is followed by 2 hours of dry blending with base to neutralize the acid. Flour products are easily reproduced to within the experimental error of the measurement of their paste viscosity (5 to 10 percent).

The new series of modified cereal flours have fluidities required for a number of industrial applications and are useful as such. Additionally, they have been modified with hydroxyalkylating agents to further broaden their potential industrial applicability.

Present emphasis of modification has been placed on tailoring flour for use as tub-sizing and coating agents in papermaking applications. The new depolymerization process obtains acceptable viscosity levels for pasted products and also maintains inherent strength characteristics of the flour to develop increases in strength properties of paper size with such materials. Evaluation studies show pastes of these flour products to be closely related in viscosity to pastes of the hypochlorite-oxidized starch but are more like hydroxyethylated acid-modified starch in hot water dispersibility and solubility. Other acid-modified flours have been prepared to give the paste viscosities required for use in textile and gypsum board industries. These latter products are now in the process of evaluation.

EVALUATION OF MODIFIED WHEAT FLOUR IN PAPERMAKING

C. R. Russell
Northern Utilization Research and Development Division

A series of chemically modified flours, including several viscosity grades of hydroxyethylated, hydroxypropylated, sulfated, and acid-modified wheat flours, were evaluated for their utility as adhesives and sizing agents for paper. Two of these modified flours, an acid-modified flour and a hydroxyethylated acid-modified flour, were found to be highly effective in improving strength properties of paper when applied as tub sizes. Bursting strength, tensile strength, and folding endurance of some papers were increased by as much as 43, 20, and 93 percent, respectively, by application of either the acid-modified or hydroxyethylated acid-modified flour. In comparative tests with some of the best commercial starch-based sizing agents, these modified flours were found to be equivalent to the commercial sizes in strength properties and about comparable to them in brightness, color (whiteness), and opacity.

Continuous sizing experiments with the hydroxyethylated acid-modified flour on small-scale laboratory equipment showed that there was little tendency for the flour components to be selectively adsorbed or for its paste to change in viscosity. On the basis of these results it is expected that hydroxyethylated acid-modified flour will perform satisfactorily in high-speed industrial sizing equipment. Thus, the major defects of unmodified flour, selective adsorption of its components and unsuitable paste viscosity, which prevent its being used in sizing operations, appear to have been overcome by a relatively simple low-cost method of modification. Preliminary evaluation of hydroxyethylated acid-modified flour as a coating adhesive indicates that this flour derivative may also perform satisfactorily in this second major outlet for adhesives in the paper industry.

PREPARATION OF LOW-PROTEIN FRACTIONS FROM SOFT AND HARD WHEAT FLOURS BY FINE GRINDING AND AIR CLASSIFICATION

V. F. Pfeifer
Northern Utilization Research and Development Division

Low-protein fractions from wheat flour are of particular interest to the Northern Division since it is hoped that they can be used directly or converted by means of physical or chemical modification into industrial products commanding large markets. Fractions containing 2.5 percent protein or less have been reported in the literature, but the published information is rather scanty. Supposedly, fractions containing less than 3.0 percent protein (often called "Industrial Starch") should be suitable for many industrial uses for which other materials are presently used.

Six commercial, unbleached, straight-grade flours were used: Pacific Northwest soft white, Michigan soft white, Indiana soft red winter, Illinois soft red winter, a hard red winter and a hard red spring. These were fractionated after fine grinding and the low-protein fractions from each were reprocessed by blending, regrinding, and reclassifying. Two of the blends were reclassified without regrinding in order to show the effect of regrinding. A sample of hard red winter flour was fractionated as-milled (without fine grinding), and the low-protein fractions were blended and reprocessed in order to determine the possible superiority of this blend. A low-protein fraction from Pacific Northwest soft white flour was reprocessed in order to yield material of the lowest protein content possible, 1.4 percent.

Fractions of minimum protein content were obtained from the softest wheat flours. The minimum protein found increased with the hardness of the wheat. Fractions containing 3 percent protein or less were separated from all soft wheat flours. The minimum protein content obtained from hard red winter wheat flour, 4.9 percent, came from the low-protein blend separated from as-milled flour. In all cases the separation of low-protein fractions from a blend was facilitated by regrinding before reclassifying.

Recovery of low-protein flour fractions having industrial potential is facilitated by selecting soft wheat flours for processing, by initially separating the material of lowest possible protein, (efficient classification), and by thoroughly regrinding and efficiently reclassifying this separated material. In some cases, selection of as-milled, instead of reground flour, may be desirable because the as-milled flour yields the minimum-protein fraction when reprocessed.

PERFORMANCE REQUIREMENTS FOR EXPANDING USES OF CEREAL-DERIVED PRODUCTS IN THE PAPER INDUSTRY

John W. Swanson
The Institute of Paper Chemistry

The principal constituent of grain is starch and while such grains also contain appreciable quantities of proteins and other materials, these products have never attained any large-scale usage in the paper industry. Therefore, this paper is concerned primarily with the starches as cereal-derived products.

It is interesting to note that starches were used for bonding and sizing papyrus as early as 3500 B.C., and have been used off and on in papermaking for the last 2,000 years. In spite of this, the technology of the use of starch in the paper industry has developed rather slowly over the last 30 years. In the past--fad, fancy, and sometimes prejudice have played an important role in determining the use of starch in papermaking. There was a time when papermakers held the opinion that starch was used only when the beater man had failed to make a good pulp. In other words, starch served to cover up his mistakes. This is certainly not true today. The use of starch in the paper industry has grown at an increasingly rapid rate until now it is one of the major raw materials for the making of many grades of paper and board. The estimated annual consumption of corn starch alone is 446,000 tons, and about 138,000 tons of other starches are used. The paper industry is second only to the food industry in the consumption of starches.

Corn starch has not always been the major starch used in the paper industry. Prior to World War II, tapioca starch, imported from the East Indies was used almost exclusively. The principal reason for this was the generally good colloidal properties of this starch. It was easily cooked in water and the dispersions were reasonably stable. Moreover, tapioca starch possesses excellent adhesive strength. Only when supplies of tapioca were cut off during the war did the industry turn to corn, wheat, and potato starches as substitutes. At first these starches were quite unsatisfactory, but as the industry learned through research and development how to prepare and use them properly, they were found to be excellent adhesives. Although tapioca is again available, it has never regained this large market in the paper industry.

The major uses of starches in the paper industry are the following:

Beater Adhesives--for improving paper strength.

Pigment Coating Adhesives--for bonding pigment-pigment and the coating to the paper and board.

Surface Sizing of Paper and Board--includes tub sizing, size pressing, and calender sizing. For controlling surface properties of paper and board such as porosity, abrasion resistance, ink-, oil-, and wax-penetration, surface fuzz, and gloss.

Laminating Adhesive--for adhering two or more layers of paper or board together for manufacture of solid fiberboard or manufacture of tubes and cores.

Corrugating Adhesives--for bonding the corrugating medium to the outside liners for the manufacture of corrugated containers. Starch has replaced almost entirely sodium silicate which was formerly used for this purpose.

Gluing Adhesives--fastening seams, end flaps in sacks, cartons, shipping containers.

A great many materials are used as beater adhesives by the paper industry both in the United States and abroad. Among these materials are the following:

1. Starches--corn, potato, tapioca, and wheat.

2. Gums and mucilages--guar, locust bean, karaya, okra, and others.

3. Modified celluloses such as methylcellulose, carboxymethylcellulose, and hydroxyethylcellulose.

4. Hemicelluloses are important potential materials for beater adhesives. However, no company supplies isolated hemicelluloses to the paper industry for this purpose.

5. Synthetic resins such as polyacrylamide is supplied to the paper industry under the name of Accostrength by the American Cyanamid Company. Other synthetic polymers of potential interest in this field are polyvinyl pyrolidone, polyvinyl acetate, polyvinyl pyridines and their copolymers. Wet-strength resins, such as urea-formaldehyde and melamine-formaldehyde, are supplied to the paper industry for the manufacture of wet-strength papers by various resin companies.

6. Synthetic latices, such as polybutadiene-styrene, neoprene, and various other rubberlike copolymers are supplied by various chemical companies to the paper industry.

Beater adhesives are used primarily for improving bonding strength between papermaking fibers. However, this benefit may be utilized in various ways, among which may be the following:

1. The improvement of bursting and tensile strengths and folding endurance, as well as tearing strength, by the addition of a

small percentage of the beater adhesive directly to the refined pulp.

2. Utilization of higher percentage of short-fibered pulps and pigment fillers at constant sheet strength.

3. Decreased refining or power requirements.

4. Increased mill capacity by speeding up pulp refining.

5. Increased production by increasing machine speed through faster draining on the wire.

6. Attainment of better fiber distribution or sheet formation through the use of beater adhesives.

7. Attainment of combinations of properties otherwise difficult to obtain in a given furnish. For example, a combination of high strength and high porosity in paper is uncommon except where beater adhesives are employed.

8. The improvement of wet strength through the addition of resins or other beater adhesives.

During recent years some of the natural gums have been replacing starches as beater adhesives and also have been reaching new markets where improved starches might be used. The reason for the growing popularity of the natural gums as beater adhesives is concerned with their superior properties in the papermaking process. It appears that the gums have somewhat stronger bonding strength per unit bonded area in paper and also improve fiber distribution which contributes to the strength of paper. Normally, beater adhesive starches are poorly retained by the papermaking fibers unless alum is used in the system. Marked improvements in this respect have been noted with the new cationic starches which appear to be substantive to the cellulose fibers. Potato starch apparently has a higher adhesive strength than corn and other cereal starches when used as beater adhesives. Further work should be done with cationic starches which have greater adhesive strength.

Large tonnages of converted starches are also used by the paper industry in the manufacture of pigment-coated paper. This product is made by applying a mixture of pigment and adhesive to paper or board at solids concentrations of 20 to 68 percent and often at very high machine speeds. The purposes of the coating are to improve printing properties, brightness, smoothness, and ink receptivity, in particular. The starches which are used for this purpose are chemically modified by several methods among which may be mentioned acid hydrolysis, dextrinization, enzymatic hydrolysis, oxidation, and

derivatization. Not all of these products are equally suitable for coating adhesives. The two least expensive, acid-hydrolyzed and dextrinized starches, have undesirable properties for certain coating purposes. Among these are the increased setback of the acid-hydrolyzed product and the poor color and low adhesive strength of the dextrinized products. Potentially, much more starch might be used for paper and paperboard pigment coating if improved starch products were available. Among the more desirable properties of converted starches which are needed at the present are the following:

1. Lower viscosity at high solids concentrations.

2. Better water-release properties.

3. Higher adhesive strength at low viscosities.

4. Greater film flexibility and elasticity for board coating.

5. Viscosity properties similar to the latices.

6. Better ink receptivity such as obtained from the more costly derivatized starches.

7. Insolubility of the starch-pigment film for corrugating and pigment-coating purposes.

Although the paper industry is growing very rapidly and it might be presumed that the use of starch would increase in the industry, there are many competing products which are displacing starches for certain purposes. It is clear that starch technology requires further research and development, both in depth and breadth, if starch usage in the paper industry is to keep pace with paper growth. A very intensive research effort might increase the rate of use of starch per ton of paper and it does not seem at all unreasonable to assume that such intensive research might lead to a 1975 market for starches which is double or even more than double the present use of the paper industry.

PERFORMANCE REQUIREMENTS FOR EXPANDING USES OF CEREAL-DERIVED PRODUCTS IN THE TEXTILE INDUSTRY

H. Y. Jennings
School of Textiles, North Carolina State College

The four major uses of starch in the textile industry, in the order of increasing importance volumewise, are as follows:

(a) Preparing printing pastes to dye fabrics, (b) stiffening and firming agent in finishing fabrics, (c) home and commercial laundries, and (d) warp sizing. These will be discussed in the above order.

(a) There are no accurate data on the amounts used in this application. The starch is mixed with dye and applied to the goods by a printing process. The dye is then set and the starch is later washed out. In this application there has always been the problem of getting sharp designs, and uniformity and consistency of color. Starch is being displaced by thermosetting resins in which the dye is dispersed. Then after application the resin is set to give the finished design. The merits of the latter process are lower cost and uniformity of printing.

(b) About 75 to 100 million pounds of starch are used to give firmness of hand. Although the starch is ultimately removed when the user washes the goods, the initial firmness has sales appeal. So-called wash and wear fabrics which the technical men refer to as easy-care or easy-handle fabrics are making inroads into this market. Recent developments in which softeners are added to make wash and wear fabrics less stiff have been an important development. Starch is still used in blue denims because stiffness is an important requirement. A similar situation exists with sheets, pillowcases, etc.

(c) Starch for home and commercial laundries is meeting competition with synthetic "starches." These are formulations of materials such as carboxymethyl cellulose, polyvinyl alcohol, and similar materials. They actually should not be called starches. This market consumes about 175 million pounds per year for laundries.

(d) In weaving yarn into fabric, it is necessary that the warp fiber (the one running the long way of the cloth) be sized or given a protective coating. Otherwise during the weaving it would become frayed and you would not get a smooth fabric. The warp fiber is sized by passing through a bath of the desired coating agent, through squeeze rolls, and then dried. The sizing bath when starch is used contains from 10 to 20 percent starch. After the cloth is woven, the starch is removed by enzymatic or chemical

treatment. The degraded starch goes into plant effluents and has created a pollution problem in practically all textile mills. This field is the largest single consumer of starch and is being seriously threatened by other materials which do not create this BOD problem.

The seriousness of this problem was illustrated by Dr. Jennings' citation of his experience with the Dan River Mills, with which he was associated previously. Four years ago the State Water Board gave them orders that by January 1, 1961, they must decrease their pollution of the Dan River by 50 percent. Their analysis indicated that 50 to 75 percent of the pollution was coming from the starch they used. One approach they studied was the installation of a purification plant. For their operation they estimated the cost of the plant would be between $500,000 and $1,000,000, and the cost of the chemicals would increase their production cost of finished goods by about 0.1 cent per yard. This would be ruinous in the low-profit textile industry. They tried a number of materials as replacements for starch and concluded that carboxymethyl cellulose (CMC) was the most practical solution. They now operate 75 percent on CMC and plan to go 100 percent to its use. They formerly used about 5 million pounds of starch per year. Conversion to CMC over starch will cost them $60,000 to $70,000 per year but is still cheaper than a disposal plant. This same trend is observed throughout the industry. A further merit of CMC as compared to starch is they get satisfactory results at 6 percent add-on compared to about 15 percent add-on with starch. This partially compensates for the higher cost of CMC.

One starch company made a trial run at Dan River Mills with a modified starch. They decreased the BOD by 80 percent and used one-third less starch for a satisfactory add-on. This material was offered at 15 cents per pound.

Dr. Jennings commented that no one in the textile industry is satisfied with the available sizes for use with synthetic fibers. In the case of glass, they use a protein formulation of unknown composition which is removed from the finished fabric by a 5 to 6 second burn-off at 1200° F. This might be an outlet for some of our wheat protein products.

Dr. Jennings concluded by stressing the importance of further research on the modification of starch which will diminish its BOD and enable it to do a more effective job. This might not only protect the current market for starch in the textile industry, but also lead to the development of new markets in the textile and other fields.

PERFORMANCE REQUIREMENTS FOR EXPANDING USES OF CEREAL-DERIVED PRODUCTS IN THE ADHESIVES INDUSTRY

Irving Skeist
Skeist and Schwarz, Inc.

Adhesives is a growth industry. Rapidly increasing amounts of bonding materials are being used in packaging; in plywood and other wood products; as binders for clay coatings on paper; as bonding agents for abrasive particles, leather, glass, and metals; as foundry core binders; in gummed and pressure-sensitive tapes. Great expansion is anticipated in particle board, laminated wood, and many other products utilizing adhesives. The adhesive bonded house is being discussed seriously.

Cereal-derived adhesives are enjoying a portion of this growth, particularly in packaging. However, the rate of growth for starches and dextrines is only 5 percent per year, while synthetic resins are increasing at least four times this percentage.

The single most important property of starches and dextrines is their low cost. In addition, they are readily available in a wide range of molecular weight, are nontoxic, have good storage life, are easy to handle, flow readily, and can have light color and good heat resistance. With the help of additives, they can be made resistant to microorganisms. Their bonding strength is adequate for paper, but leaves something to be desired with many other adherends.

Of their shortcomings, the most troublesome is their lack of water resistance. In addition, it would be desirable if they had a better combination of bonding strength, mileage, setting speed, tack, solids content, and flexibility, especially at low temperatures.

If water resistance could be improved notably, there might be increased opportunities for cereal-derived adhesives in some types of wood bonding, and to a greater extent in packaging applications. This is a difficult problem, but the potential gains justify a major effort. The problem can be best attacked by converting the polysaccharide to a form that is insoluble in water. There are two fundamentally different ways to accomplish this. First, the starch or dextrine can be reacted with a cross-linking agent, converting all the separate molecules into one big molecule. Second, the polysaccharide can be modified so that the individual molecules are less sensitive to water.

Of the cross-linking agents, formaldehyde is the best known, either by itself or in such a combination with urea as

dimethylolurea. But what about other means of tying polysaccharides to one another? Acrolein, now under investigation at Peoria, has the advantage of providing a different type of group, the vinyl group, for further reaction. Why not phenol-formaldehyde and resorcinol formaldehyde? Might these confer the same insolubility to starch that they give to its sister molecule, cellulose? A variety of other difunctional or polyfunctional reactants are worth investigating.

The other technique that would seem promising is the incorporation of water-resistant groups into the starch molecule. The partial esterification of starch with fatty acids would help to take care of two surpluses at once. In addition to helping the molecule shed water, the fatty acid groups might make it more flexible. If unsaturated fatty acids were used, there would be again a site for further reactions, including cross-linking if desired.

Graft polymerization offers still another means of modifying the solubility and performance of the starch molecule. The USDA is sponsoring research in this direction.

Among the vast number of other reactions and reactants that might have some value for starch are the peptones and other protein-hydrolysates. Perhaps it is possible to make a hybrid molecule that combines the best advantages of the polysaccharides and the proteins.

LIPIDS AND LIPOPROTEINS IN WHEAT FLOURS

G. O. Kohler
Western Utilization Research and Development Division

The presence of complexes of lipid and protein constituents in flour, dough, and gluten has been repeatedly substantiated by various kinds of evidence since the presence of 5 to 15 percent lipid in gluten was first pointed out by Dill about 35 years ago. Flour lipids are also known to modify baking characteristics of flours. However, information on the nature of lipoprotein components in flour or on the specific lipid and protein components involved in the formation of protein-lipid complexes in doughs or gluten is still little more than fragmentary.

Current work at WU includes examination of the material extracted by water-saturated n-butanol for protein and lipoprotein constituents. By treatment of such extracts with ethanolic HCl (as used by Balls, Hale and Harris in the isolation of purothionin to disrupt lipid-protein complexes), protein material containing appreciable disulfide-sulfur is separated, but it appears to differ from purothionin. Alternatively, intact lipoproteins have been separated by partition between petroleum ether and water; electrophoretic examination of the water-soluble material shows predominantly one component, with a mobility approximately that of the alpha component of gluten in aluminum lactate buffer. The turbidity of the solutions interfered with interpretation of the results.

The investigations of lipid constituents have been assisted greatly by the availability of recently developed techniques, particularly countercurrent extraction, silicic acid column chromatography, and gas chromatography. Current work at WU based on silicic acid column chromatography concerns separations and characterization of phospho- and glyco-lipid components from flour oil. Separations of simple from complex lipids have been obtained for a hard red spring wheat flour and its gluten, together with segregation of the complex lipids into five classes of components. One class contains a lipoprotein component; others contain the galactolipids, phosphatidylethanolamine and phosphatidyl choline.

Lipids from high- and low-protein air-classified fractions of a commercial soft wheat flour were compared with those of the original flour. Total lipid content increased with protein content, though divisions into simple and complex lipids were similar for all three flours. Complex lipids from the high- and low-protein flours, fractionated on silicic acid, showed generally similar distributions. These were also similar to results from hard red spring wheat flour.

DISTRIBUTION OF ALBUMIN AND GLOBULIN PROTEINS IN AIR-CLASSIFIED FRACTIONS OF A SOFT WHEAT FLOUR

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Apart from their commercial importance, fine-grinding and air-classification of wheat flours provide a unique opportunity for research on relationships between composition and baking performance. From the same starting material fractions having different properties may be obtained that are free of complicating factors associated with crop year, growth location, and the like. The present study is concerned with preliminary investigation of the protein composition of a series of fractions obtained from a club wheat flour by these new milling methods.

A pastry flour derived from Omar variety wheat mixed with a small amount of Brevor variety was separated into a coarse fraction and a fine fraction by air-classification. The fine fraction was then mixed with about twice its weight of original flour and the blend reground in a pin mill. The reground blend was separated into eight fractions by air-classification. The various fractions were then blended in a way to give three flours of 11.5 to 12.0 percent protein for bread test-baking, seven flours of 6.5 to 7.5 percent protein suitable for bleaching and test-baking in white cakes, and four flours of 7 to 9 percent protein appropriate for testing as cookies.

All blends and starting materials were analyzed for total albumin protein and total globulin protein by a chemical method dependent on the total protein nitrogen, amide nitrogen, and tryptophan nitrogen of purified preparations of the proteins. Values obtained for the original flour are typical for flours milled from club and other low-protein soft wheats. The high-protein fractions, rich in wedge protein, tend to be lower in soluble protein than the low-protein fractions, rich in adhering protein. However, the ratio of albumin to globulin proteins is about the same for the original flour and these fractions. The very low albumin to globulin ratios obtained are consistent with the very poor bread-making quality of the flour and its fractions and with results obtained in previous research on other flours. No correlations between protein composition and cake or cookie baking quality were discernible.

FUNDAMENTAL ASPECTS OF WHEAT VARIETY EVALUATION

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The largest single use of wheat is for human food. Therefore, it is of the utmost importance that high standards of quality be maintained in our wheat and flour, and that such standards be constantly appraised for suitability to meet current and future demands.

State, Federal, and private wheat breeders released 45 new wheat varieties to U. S. farmers during the 5-year period 1954 to 1959. Several varieties not known to farmers 5 years ago now dominate the acreage in their respective market classes. Hence, in many instances, millers cannot get the varieties available to them 10 or even 5 years ago. A bright note here is the fact that numerous varieties specified as undesirable and discounted in the Government's price support program have declined and disappeared.

It should be apparent that, unless we are willing to leave quality to chance, an extensive program of testing new varieties is required. Along with this there must be an understanding of the basic elements or components of quality. This is the twofold job of the four wheat quality laboratories our Service maintains, one for each major wheat-producing region.

A few recent developments in our research, in addition to variety evaluation, have been selected for discussion this year. No attempt is made to give a full account of the work underway.

Protein content as a distinct varietal characteristic is receiving renewed recognition and emphasis. Except for minor differences, most varieties produce similar protein levels when environmental and nutritional factors are equal. In contrast, however, is a genetic source of 1 to 3 percent more protein discovered several years ago in hybrids involving two Brazilian wheats with soft red and hard red wheats. Air-classification work has been initiated on flour from some of these experimental varieties, and the quality of the gluten is to receive intensive study.

The quality of protein in hard winter wheats continues to run highest for breadmaking when the grain is cut at the time the natural moisture drops to about 40 percent. This is 8-10 days before wheat may be combined.

Washing experiments with Sentry durum have shown that the gluten is nonelastic, soft, slimy, and sticky. Recently, however, extraction of certain components, not identified chemically,

resulted in formation of a firm, elastic gluten similar to that from Langdon. This may help evaluate varieties more precisely for canning, noodles, and macaroni than has previously been possible.

Bread quality testing work, especially with hard red spring wheat, has achieved new high levels of precision through the use of the "remix method." Differences in strength between varieties are magnified in the bread-baking test and varieties are more readily ranked in order of physical dough characteristics as well as loaf volume.

Cake-baking methods have long been a matter of controversy. The lean formula, which eliminates milk and egg whites, is gaining in usefulness for layer cake testing because it is sensitive to many component differences found in flours, such as liquid-carrying capacity, bleach treatment, particle size, patent extraction, and protein content. The test correctly ranks varieties according to long-standing commercial experience. The ratios of basic cake formula ingredients--baking powder, shortening, flour, sugar, and water--have been studied by means of the Box-Wilson multiple response surface type of experimental design. The effect of baking powder and sugar-water ratios was curvilinear and critical; a strong interaction effect resulted in sharply reduced volumes if these ingredients were out of balance. The effect of shortening was linear and had little effect on volume within the range studied. Larger-than-normal cakes resulted from increasing the ratio of flour to sugar plus water.

Cookie quality is closely related to the hydration capacity of flour. In addition to hydration due to starch and protein, it has been found that hydrophilic components in the starch tailings have a bearing on quality. Heating of good- and poor-quality doughs proceeds at the same rate in baking and liquid losses are similar. However, viscosity increases sooner and at lower temperatures of the heating process in poor-quality doughs.

Micro tests were developed to utilize the meal from the 5-gram mill. Loaf volume and cookie quality were predicted from a series of micro tests of the relation of the gas-retention properties of an extracted and unextracted flour. Mixing time and water absorption were also determined.

Air-classification work on five wheat varieties indicates that, regardless of where grown, typical soft varieties give the greatest protein shift, with higher yield of the fine fraction and higher protein content than Purkof, a semihard variety. Pearling test results correlated more highly with protein shift than particle-size index or break-flour yield.

SEPARATION OF WHEAT GLUTEN PROTEINS BY STARCH GEL ELECTROPHORESIS

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The question of wheat gluten heterogeneity has only recently been resolved by the electrophoretic studies of Jones, Taylor, and Senti. Through the use of buffers of low pH and ionic strengths, these workers were able to demonstrate the presence of four major and one minor component in Ponca wheat gluten using the Tiselius method of electrophoresis. The possibility of detecting additional components by further varying conditions or by using other forms of electrophoresis then was considered.

Zone electrophoresis has theoretical advantages over moving-boundary electrophoresis and has given superior resolution of other proteins with starch gels as the supporting medium. Starch gel electrophoresis in the presence of concentrated urea has proved applicable to wheat gluten proteins. Through the use of this method we were able to demonstrate the presence of eight components, two of which were not previously detected. The absence of any dissociative effect of urea on the gluten proteins was demonstrated by comparisons of patterns obtained with and without urea.

A water-soluble protein fraction (albumins and globulins) isolated from a gluten preparation, showed nine components when examined by starch-gel electrophoresis in the presence of concentrated urea. This new analytical method should find wide application in further studies of wheat-gluten proteins--especially in studies to compare proteins from different varieties of wheat. It also will provide an additional criterion of purity for components isolated during structural studies.

EFFECT OF DISULFIDE BOND SPLITTING ON MOLECULAR SIZE OF α_1-GLUTEN

H. C. Nielsen
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Alpha-1 gluten is the fastest moving electrophoretic component of gluten. It is isolated from whole gluten by a pH-ionic strength procedure involving adding sodium acetate to a 0.3-percent solution of gluten in 0.1 N acetic acid to form a 0.05-ionic strength, pH 4.5 buffer which causes α_1-gluten to precipitate. The remainder of the proteins, termed "soluble fraction proteins," are then precipitated by adding M sodium chloride and adjusting the pH to neutrality.

The unique viscoelastic properties of gluten reside in its α_1 component. Evidence for this is that α_1-gluten will form a typical gluten ball while "soluble fraction proteins" will not. Alpha-1 gluten has a high intrinsic viscosity which is markedly reduced by disulfide-bond-splitting agents. This is strongly reminiscent of how disulfide-bond-splitting agents cause a dough or a gluten ball to relax and fall apart. "Soluble fraction proteins" have a low intrinsic viscosity.

The disulfide bonds of α_1-gluten were cleaved by oxidation with performic acid at 0° C. Performic-acid-oxidized α_1-gluten had one major electrophoretic component in a buffer containing 0.0167 M aluminum lactate adjusted to pH 3.0 with lactic acid. Cleaving the disulfide bonds of α_1-gluten caused the protein to completely lose its viscoelastic properties.

A major difficulty in studying the molecular weight of α_1-gluten by sedimentation analysis is lack of solubility. Alpha-1 gluten is only soluble at extremes of pH and at very low ionic strength, its solubility being enhanced by hydrogen bond-breaking agents like urea. For sedimentation analysis a protein should not only be dissolved but dissolved so that it exhibits its minimum molecular weight. Also, this method is best carried out in an aqueous solution of some ionic strength and near the isoelectric pH of the protein under study. After several experiments a suitable system was found for the sedimentation analysis of α_1-gluten. This was an aqueous system containing 0.03 M ammonium hydroxide and M urea. A little EDTA was added to this system, in the case of α_1-gluten, to sequester heavy metal ions which would otherwise catalyze some disulfide bond cleavage.

Sedimentation analysis was carried out by a method suggested by Trautman [J. Phys. Chem. 60:1211 (1956)] which gives molecular weight and degree of polydispersity from ultracentrifuge runs made at various speeds. Results showed that α_1-gluten was a mixture of sedimenting species with molecular weights ranging from about 50,000

up into the millions. The weight-average-molecular weight of α_1-gluten is 1.6 million. Cleaving the disulfide bonds of α_1-gluten reduced it to a single sedimenting species with a molecular weight of 21,000. This is thought to be the basic peptide unit of α_1-gluten.

To answer a question on the lipid content of α_1-gluten and one on the treatment used to make gluten starch-free: Whole gluten used in this study was isolated from Kansas-grown Ponca wheat flour by the procedure of Jones, Taylor, and Senti [Arch Biochem. Biophys. 84: 363 (1959)]. This involved extracting the flour with dry n-butanol to remove lipid materials; forming a dough ball and washing out starch with 0.1 percent sodium chloride; dispersing the gluten in dilute acetic acid with the aid of a high speed blender; centrifuging first at low speed than at 20,000 g. to remove starch; heating to 98° C. to inactivate proteolytic enzymes; then freeze-drying.

To answer the question about whether or not gliadin goes with the "water-soluble proteins": Alpha-1 gluten and glutenin have identical electrophoretic patterns in aluminum lactate buffer. Therefore they may be identical. By a process of elimination it might be concluded that gliadin and "water-soluble proteins" may be the same. However, Holme and Briggs [Cereal Chem. 36:321 (1959)] claim that gliadin will form a gluten ball while the "water-soluble proteins" will not. It might be added that electrophoretic analysis of gliadin in aluminum lactate buffer shows that it contains a fair amount of α_1-component.

DISCUSSIONS

Morning Session:

Discussions following each talk were very limited because of the lack of time.

Where speakers referred to the use of starch, the potential use of flour should be inferred.

Research on the chemical modification of flour has been principally on straight grade flours from soft and hard red winters. No controlled experiments on the effects of particle size of the flour modified have been made. Dispersibility has been satisfactory in comparison with the reference starches 5591 and Superfilm 40.

Just initiated is research on the effects of conditioning wheat on its milling performance, using the Brabender mill and microscopic and histochemical evaluation. Later studies will extend this work to flours milled with the Miag mill and to air-classified flours. Variations in protein shift with air-classification found to date have been more dependent on variety than on location grown. Pacific Northwest wheats studied so far and for which reports are being prepared include Omar, Brevor, Baart, and Rio.

Afternoon Session:

The effects of particle size on baking qualities are very important but we have little data from controlled experiments for relating causes and effects. In the air-classification of Midwest varieties, Fairfield and Thorne have shown the greatest protein shift.

In his concluding remarks C. L. Mast, Jr. reaffirmed Mr. Schopmeyer's position that our need is for completely new industrial uses for flour based on the properties revealed by fundamental research.

UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service

Northern and Western Utilization Research and Development Divisions
and Crops Research Division
with the
Technical Advisory Committee, Millers' National Federation, and
Technical Committee, Association of Operative Millers

PROBLEMS AND OPPORTUNITIES IN THE INDUSTRIAL UTILIZATION OF WHEAT
Peoria, Illinois
October 4, 1960

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